इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

IS 1448-50 (1977): Methods of test for petroleum and its products, Part 50: Chlorine in new and used lubricants (sodium alcoholate method) [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

“ज्ञान से एक नये भारत का निर्माण”
Satyanarayan Gangaram Pitroda
“Invent a New India Using Knowledge”

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartṛhari—Nītiśatakam
“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

IS : 1448 [P : 50] - 1977
(Reaffirmed 2006)
Adopted : 7 November 1977

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 50]

CHLORINE IN NEW AND USED LUBRICANTS (SODIUM ALCOHOLATE METHOD)

(*Second Revision*)

First Reprint JULY 1988

UDC 621·892:543:546·13

Adapted, with permission, from the Joint Publication ASTM D 1317-64 (Reapproved 1973)/IP 118/65

1. SCOPE

1.1 This method describes a procedure for the determination of chlorine content of lubricating oils and greases with which chlorinated additives have been blended. It is equally applicable to new or used oils and greases and to the additive concentrate. The procedure assumes that compounds containing halogens other than chlorine will not be present.

2. OUTLINE OF THE METHOD

2.1 The sample, dissolved in a low boiling hydrocarbon mixture, is boiled under reflux with metallic sodium and *n*-butyl alcohol. Under these conditions the chlorine is converted to sodium chloride which is then extracted with water. The chloride in the extract is determined volumetrically by titration with silver nitrate in the presence of thiocyanate.

3. APPARATUS

3.1 Reaction Vessel — consisting of a 300-ml round-bottom flask with standard-taper joint and surmounted with a water-cooled condenser (*see* Note).

NOTE — The condenser tube shall be wide enough to allow rapid reflux without hold-up.

© *Copyright* 1978

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

February 1978

4. REAGENTS

4.0 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS:1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.1 Benzyl Alcohol — The benzyl alcohol used shall not show a visible precipitate when tested with alcoholic silver nitrate ($AgNO_3$) solution.

4.2 *n*-Butyl Alcohol — *See* IS:361-1973†.

4.3 Ferric Alum Indicator Solution — Dissolve 350 g of ferric alum $[Fe_2(SO_4)_3.(NH_4)_2SO_4.24H_2O]$ in 1 litre of water and add 200 ml of aerated nitric acid (1:2).

4.4 Isoamyl Alcohol — The isoamyl alcohol used shall not show a visible precipitate when tested with alcoholic silver nitrate ($AgNO_3$) solution.

4.5 Naphtha — paraffinic, distilling between 99 and 121°C.

OR

Petroleum Hydrocarbon Solvent (100/120) — *See* IS:1745-1966‡.

4.6 Nitric Acid (1:7) — Mix 1 volume of concentrated nitric acid (relative density 1·42) with 7 volumes of water. Aerate to remove oxides of nitrogen.

4.7 Phenolphthalein Indicator Solution (10 g per litre) — Dissolve 1 g of phenolphthalein in *iso*-propyl alcohol (60 percent) and dilute to 100 ml with the alcohol.

4.8 Standard Silver Nitrate Solution (0·1 N) — Dissolve 16·989 0 g of dry (1 hour at 110°C) silver nitrate ($AgNO_3$) in water and dilute to 1 litre in a volumetric flask with water.

4.9 Sodium Metal

4.10 Sodium Hydroxide Solution (80 g per litre) — Dissolve 80 g of sodium hydroxide in water and dilute to 1 litre.

4.11 Standard Thiocyanate Solution (0·05 N) — Dissolve 3·80 g of ammonium thiocyanate (NH_4CNS) or 4·85 g of potassium thiocyanate (KCNS) crystals in water and dilute to 1 litre. To standardize this solution, measure 40 ml into a flask containing 5 ml of nitric acid (1:7) and 50 to 60 ml of water. Swirling the flask constantly, measure 22 to 24 ml of

---

*Specification for water for general laboratory use (*second revision*).

†Specification for normal butyl alcohol, technical (*second revision*).

‡Specification for petroleum hydrocarbon solvents (*first revision*).

0·1 N silver nitrate solution from a burette; then stopper and shake for 20 to 30 seconds. Add 2 to 3 ml of ferric alum indicator solution and titrate with the thiocyanate solution to a red end point. Calculate the normality of the thiocyanate solution as follows:

$$\text{Normality of thiocyanate solution} = \frac{A\,N}{B}$$

where

A = volume in ml of standard silver nitrate solution used,

N = normality of the silver nitrate solution, and

B = total volume in ml of the thiocyanate solution used.

5. PROCEDURE

5.1 Shake the sample, if liquid, to ensure homogeneity. Weigh a portion, estimated to contain 0·05 to 0·1 g of chlorine, into the 300-ml flask. Add 25 ml of naphtha or petroleum hydrocarbon solvent until the sample is dissolved.

5.2 Reaction and Extraction

5.2.1 To the solution of the sample, add 5 ml of *n*-butyl alcohol and 2·8±0·2 g of freshly cut sodium. Connect the flask to the condenser and boil the liquid vigorously under reflux for 1 hour. Introduce an additional 20 ml of *n*-butyl alcohol through the top of the condenser and continue refluxing until no metallic sodium remains.

5.2.2 Add 25 ml of water to the flask through the condenser, cool to room temperature, and transfer the mixture to a 500-ml separatory funnel. Wash the flask with 50 ml of nitric acid (1:7) and then with 50 ml of naphtha or petroleum hydrocarbon solvent. Add these washings to the separatory funnel. Adjust the acidity of the aqueous layer until just acidic phenolphthalein, using nitric acid (1:7) or sodium hydroxide solution for this purpose; then add 12 to 13 ml of nitric acid (1:7).

5.2.3 Shake the funnel and contents, and, after settling, draw the aqueous layer into a clean 500-ml glass stoppered conical flask. Wash the original 300-ml reaction flask with 25 ml of water, transfer to the separatory funnel, wash the naphtha or petroleum hydrocarbon solvent layer which remained in the separatory funnel after separating the aqueous layer; transfer this washing to the 500-ml flask. Repeat with a second 25-ml portion of water.

5.3 Chloride Determination

5.3.1 Boil the combined acidic aqueous extracts and washings until free of hydrogen sulphide as detected by dipping a piece of lead acetate test paper into the solution (no black discolouration). If the solution is clear, proceed with the directions in **5.3.2** and **5.3.3**; if not, filter the solution through a qualitative filter paper into another 500-ml glass-stoppered conical flask. Rinse the original flask with two 25-ml portions of water, using each portion to wash the filter paper.

5.3.2 After the solution has attained room temperature, add 10 ml of *iso*amyl alcohol or benzyl alcohol (*see* Note); 3 ml of ferric alum indicator and, from a burette, 0·4 to 0·6 ml of 0·05 N thiocyanate solution. Swirling constantly, titrate with 0·1 N silver nitrate solution until the red colour is discharged; then add 2 to 5 ml in excess.

NOTE — Because of the extreme toxicity of nitrobenzene, its use has been discontinued, and the use of either *iso*amyl or benzyl alcohol is recommended. Since both of these alcohols are considered to have very moderate toxic properties, care should be exercised with their use, but they are relatively many times less toxic than nitrobenzene.

5.3.3 Stopper the flask tightly and shake vigorously for 15 seconds to coagulate and remove the precipitate from the aqueous phase [CAUTION: *see* Note]. Without refilling the burette, titrate slowly with 0·05 N thiocyanate solution until the end point is approached, as indicated by a more slowly fading red colour. Stopper the flask, shake vigorously for 20 to 30 seconds, and continue the titration until one drop produces a distinct reddish colouration that does not fade upon swirling or vigorous shaking.

NOTE — *CAUTION* — Care should be exercised in opening the flask after shaking. Pressure may develop, causing small amounts of the acid solution to be sprayed from the mouth of the flask as the stopper is removed.

5.4 Blank Determinations — Simultaneously with each set of sample determination, make duplicate blank determinations, following exactly the procedure use for the sample determinations (except for omission of the sample). Always use identical reagents for the blank determination and for the sample determination. If the titration exceeds 0·05 ml, discard the responsible reagent.

6. CALCULATION

6.1 Calculate the total chlorine content of the sample as follows:

$$\text{Chlorine, percent by mass} = \frac{(A\,N - B\,N_1) \times 35{\cdot}46}{10\,M}$$

where

A = volume in ml of standard silver nitrate solution used,

N = normality of the silver nitrate solution,

B = volume in ml of thiocyanate solution used,

N_1 = normality of the thiocyanate solution, and

M = mass in g of the original sample used.

7. REPORTING

7.1 Report the result to the nearest 0·01 percent as the chlorine content.

8. PRECISION

8.1 Results of duplicate tests shall not differ by more than the following amounts:

Chlorine Content, Percent	*Repeatability*	*Reproducibility*
0·1 to 1·0	0·03	0·04
Over 1·0	3 percent of amount present	6 percent of amount present

Reprography Unit, BIS, New Delhi, India